



MISSOURI DEPARTMENT OF TRANSPORTATION
PROJECT OPERATIONS
Jefferson City, Missouri
Test Method
MoDOT T21

DETERMINATION OF COBALT IN TUNGSTEN CARBIDE INSERTS

1.0 SCOPE

- 1.1 This method describes a procedure for determining the percent of Cobalt in Tungsten Carbide Inserts used in Snow Plow Blades, using Atomic Absorption Spectrophotometry

2.0 EQUIPMENT AND REAGENTS

- 2.1 An Atomic Absorption Spectrophotometer.
- 2.2 Hydrofluoric Acid (HF), 48 percent
- 2.3 Nitric Acid (HNO₃), specific gravity 1.42
- 2.4 Hydrochloric Acid (HCl), specific gravity 1.19
- 2.5 Distilled Water

3.0 PREPARATION OF STANDARDS

- 3.1 1000 ppm Cobalt stock solution: This solution can be purchased from a number of sources, or it can be prepared in the laboratory from a suitably pure Cobalt salt.
- 3.2 8.00% Cobalt standard solution: Place approximately 250 mL distilled water into a 1000 mL volumetric flask. Add 10 mL HF, 5 mL HNO₃, and 20 mL HCl. Pipette 20.00 mL of 1000 ppm Cobalt stock solution into the flask and dilute to volume with 1:99 HCl prepared with distilled water. This solution is equivalent to 8.00% cobalt.
- 3.3 16.00% Cobalt standard solution: Place approximately 250 mL distilled water into a 1000 mL volumetric flask. Add 10 mL HF, 5 mL HNO₃, and 20 mL HCl. Pipette 40.00 mL of 1000 ppm Cobalt stock solution into the flask and dilute to volume with 1:99 HCl prepared with distilled water. This solution is equivalent to 16.00% cobalt.
- 3.4 Blank solution: Prepare a blank by the same procedure used to prepare the 16.00% Cobalt standard, omitting the addition of 40 ml of Cobalt stock solution.

4.0 PROCEDURE

- 4.1 Crush or grind an insert until approximately 4-5 grams will pass a No. 50 sieve. Weigh, to 0.1 mg, 0.2500 grams of the material passing the No. 50 sieve, and place in a



platinum crucible having a volume of 25-35 mL. Add 10 mL of HF and 2 or 3 drops of HNO_3 . Cover the crucible immediately and heat gently. Add 2 or 3 drops of HNO_3 each time the reaction subsides, keeping the crucible covered as much as possible. When solution of the sample is complete, remove the cover, rinse it with H_2O , and cool to room temperature. Place approximately 100 mL of distilled H_2O in a 400 mL beaker and quantitatively transfer the solution into the beaker. Rinse the crucible 4-5 times with distilled H_2O , once with 1:99 HCl, and finally with distilled H_2O . Add 20 mL HCl to the contents of the beaker and dilute to approximately 250 mL with distilled H_2O . Cover with a watch glass, boil vigorously about 5 minutes, digest 3 hours at low heat, and let precipitate overnight. Filter on Whatman No. 42 paper into a 1000 mL volumetric flask, washing 10-12 times with hot 1:99 HCl. Cool and dilute to volume with 1:99 HCl. (Note: For inserts with higher concentrations of cobalt, the dilution should be increased to allow for the cobalt concentration to fall within the prepared standard solution range.)

4.2 Calibrate the instrument using the blank solution, the 8.00% standard solution and the 16.00% standard solution, then determine the concentration of the sample solution.

5.0 CALCULATION AND REPORT

5.1 The method of calculating the percent Cobalt in the sample will vary with the make and model of instrument used. Report the Cobalt in the sample to the nearest 0.1% as follows:

% Cobalt (Co)

